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Langmuir Probe And Optical Emission Studies Of Ar, O_2 and N_2 Plasmas Produced By An ECR Microwave Source

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Abstract

An understanding of electron cyclotron resonance microwave plasma discharges requires the measurement of plasma parameters. In argon, nitrogen and oxygen plasmas, cylindrical Langmuir probes were used to determine the variations of plasma parameters (ion density, N_i, floating potential, V_f, plasma potential, V_p, and electron temperature, T_e) with applied power (100-300 W) and pressures ($1\text{-}10\times10^{-4}$ Torr). Simultaneous optical emission (OE) spectra were also obtained, and, using knowledge of excited and neutral peak intensities and transition energies, the electron temperatures were calculated. By the use of the Langmuir probe electron temperature results, the electron temperatures obtained from OE resulted in scaling factors, C, of 2 $eV^{-.75}$, .15 $eV^{-.75}$, and 2 $eV^{-.75}$ for argon, oxygen, and nitrogen, respectively.

I. Introduction

Electron cyclotron resonance (ECR) plasma sources have been implemented for use in both deposition and etching applications (1-3). These sources are attractive because of the high degree of ionization they offer and because high ion currents can be maintained, even at low processing pressures (< 1 mTorr) and low ion energies.

The characterization of these ECR sources in plasma processing situations has mainly focused on the use of Langmuir probes, and their use has been widely documented (4-6). However, Langmuir probes are subject to contamination in oxygen or halogens, where the discharge specie can react with the probe tip. Also, probe results do not yield information about the concentrations of the reactive neutral species that are known to be important in many deposition and etching processes.

Optical emission spectroscopy, OE, has been used as a general diagnostic tool for a wide variety of plasma processes (7-10). Unlike Langmuir probe techniques, optical emission is non-invasive and is capable of detecting both neutral and ionic species. Optical emission can be used to measure absolute electron temperatures, T_e, with knowledge of transition intensities, transition probabilities, and other factors (7). However, to simplify the necessary calculations, optical emission can be used to measure variations in electron temperature; these values are then placed on an absolute scale by comparison with Langmuir probe electron temperature values (8).

This paper presents results of Langmuir probe and optical emission experiments performed under the same experimental conditions. The T_e information obtained using both of these diagnostic methods will be compared, and the scaling factors, C, that relate the two techniques are reported. Plasma discharges of argon, oxygen, and nitrogen were investigated, argon due to its inert nature, and the latter gases because of their importance in plasma deposition reactions.

II. Experimental Apparatus

A schematic of the experimental apparatus is shown in Figure 1. The chamber is evacuated by a diffusion pump (1200 l/sec) and a roughing pump (12 l/sec). Gas flows were controlled by mass-flow controllers; pressures were measured by an ionization gauge and a capacitance manometer.

The microwave power is continuously variable from 100 to 400 W and is at a frequency of 2.45 GHz. Reflected power is monitored by a directional coupler and a triple stub tuner is used to impedance match the applied microwave power into the plasma. The microwave power is introduced by a rectangular waveguide operating in the TE_{10} mode into a cylindrical cavity surrounding the resonance region.

A set of two solenoids surround the cavity where the plasma is produced. The independently powered field coils provide a static magnetic field that is used both to create the resonance condition for efficient plasma absorption and to extract it to where it can be used.

The Langmuir probe measurements were performed using a tungsten wire (diameter = 0.22 mm) that protruded 0.5 cm from a quartz sleeve, and was mounted on a linear motion feedthrough so that measurements could be taken along the chamber axis. A bias voltage, in most cases from -50 V to 50 V, was applied to the probe and the subsequent current from the plasma was measured using an electrometer.

All measurements were taken with the probe at 1.7 cm from a pseudo sample stage, which replicates the stage to be used in future depositions except that it has a hole in the middle through which the probe may pass. The distance between the stage and ECR source is 18 cm.

The optical emission experiments were performed in conjunction with the probe experiments using a 3 mm liquid light guide which transmitted plasma emission light from a viewport on the plasma chamber to the entrance slit of a 0.22 m monochromator. The viewport was located parallel to the stage position. Wavelength scans from the 350 to 800 nm region were obtained.

III. Langmuir probe

Langmuir probe I-V curves contain three distinct regions that correspond to the collection of charged particles of different representative energy (11). In Figure 2a, typical I-V curves for argon at two pressures are shown. At negative probe bias, called the ion saturation region, all of the electrons are repelled from the probe, and the slope of the probe I^2-V curve (4) yields:

$$N_i^2 = -\frac{4\pi m_i}{3A^2e^3}\left(\frac{\partial I_i^2}{\partial V_p}\right) \tag{1}$$

where N_i is ion density, m_i is ion mass, and A is probe area. From this, plasma density can be determined from the ion current. The plasma densities for all three gases were in the 10^9 ions/cm^3 range and are plotted as a function of pressure in Figure 3. Since plasmas are electrically neutral, the plasma density is given by and equal to both ion density and electron density. It is important to add that if the microwave excitation energy were to reach the probe, erroneously low plasma densities would be measured (5). In order to determine whether we have such a situation, we used our Langmuir probe to measure the plasma density profile up to the mouth of the plasma source, and found it to be greater than 3×10^{10}/cm^3 near the source. If our probe was effected by the microwaves, this value would be lower than the true value. The plasma density in the plasma source should be higher than the critical density value of 7.5×10^{10}/cm^3 for 2.45 GHz (12). Thus, we are reasonably certain that the effect of microwaves on our reported plasma density measurements is negligible. In the intermediate region of the curve, which extends from the point where the probe current is zero (the floating potential) to where the slope of the curve begins to decrease, both ions and electrons can reach the probe. In the transition region, the slope of the probe ln(I)-V curve gives the relationship:

$$\frac{1}{T_e} = \frac{\partial \ln(I_e)}{\partial V_p} \tag{2}$$

for a determination of the electron temperature (4), where I_e is electron current. Ln(I)-V plots for argon at two pressures, shown in Figure 2b, reveal the differences in plasma potential and electron temperature caused by changing pressure. Measured electron temperatures for argon, oxygen, and nitrogen were 6-12 eV, with electron temperature decreasing with pressure. To determine the plasma potential (the point where random ion and electron currents are unaffected by the bias voltage and reach the probe), a linear fit was assigned to the probe data at the beginning of the electron saturation region, which is the final region of the curve where only electrons are collected by the probe. The plasma potential was then defined as the "knee" of the lnI-V curve, or the intersection of the electron saturation region with the transition region. Other researchers (13) have evaluated this method, and report that it yields results systematically consistent with other plasma parameters. The plasma potentials for all the gases varied from 27 to 15 V, again decreasing with increasing pressure.

In the oxygen environment, a problem of oxide build-up on the probe was encountered which caused alterations in the current-voltage curves after ~15 min of exposure under positive bias. The probe then required cleaning to eliminate the oxide. For the cleaning, the probe was kept at a negative bias (-50 V) in an argon plasma for several minutes; by allowing only an ion flux to bombard the probe surface, the contamination layer can be sputtered away. To minimize this contamination problem, the experimental data was collected quickly. Each run took under 5 minutes, and, in between runs, the probe was kept at a negative bias.

IV. Optical emission

Spectroscopic diagnostics can be made by use of the intensities of plasma emission lines (14,15,16). In the Corona plasma model, the ionizational equilibrium is a balance between the processes of collisional ionization (and excitation) and radiative recombination (and spontaneous decay). Because the densities of our plasmas are ~10^{10} /cm^3, the Corona model is an appropriate one. Griem (15) provides an equation suitable for use with a Corona plasma model:

$$\frac{I'}{I} \approx \frac{f'g'\lambda^3}{fg\lambda'^3} \exp\left(\frac{E_\infty - E' - E_\infty + E}{kT}\right) \frac{S}{\alpha} \tag{3}$$

where primed quantities refer to the line from the higher ionization stage, I λ, g, and, f are total intensity, wavelength, statistical weight (of the lower state of the line), and absorption oscillator strength, respectively, of one line, E is excitation energy and E_∞ is the ionization energy. Applying Griem's equation gives the ratio of intensities proportional to S/α, where S is an ionization factor and α is a recombination factor. A functional form must be assigned to the factors S and α, which was done by Cox (16) with reference to Huddlestone and Leonard (17), who suggest that S has a $T_e^{0.25}$ dependence, and that α has a $T_e^{-0.5}$ dependence.

Plasma de-excitation occurs by radiative transitions. The intensity ratio of emission lines derived from transitions between levels i-x in the excited singly charged ion ($I^{+}_{i,x}$) and p-y in the excited atom ($I^{0}_{p,y}$) can be related to the electron temperature as follows (8):

$$\ln\left(\frac{I^{+}_{i,x}}{I^{0}_{p,y}}\right) = \ln C + 0.75 \ln T_e - \left(\frac{E_g + E_{i,g} - E_{p,g}}{T_e}\right) \tag{4}$$

where k is the Boltzmann constant, T_e is the electron temperature, E_g the ionizational potential of the atom, $E_{i,g}$ the excitation energy of the singly charged ion from its ground state (g) to level i, and $E_{p,g}$ the excitation energy of the atom from ground state to level p. The scaling factor, C, depends on i, x, p and y, and is a complicated expression involving unknown collisional cross-sections (8,15). Under certain conditions (eg, same gas, pressure range), C may be treated as a constant, and can be evaluated using line intensities from optical emission data in association with the T_e's determined with a Langmuir probe.

V. Results

The Langmuir probe ion density results for argon, oxygen and nitrogen in Figure 3 show a decrease in ion density with increasing pressure. The plasma potentials also decrease with pressure, and are between 15 and 26 V, as can be seen in Figure 4. The information on ion densities and plasma potentials determined from the probe, and the electron temperatures determined from both methods, which are discussed below, are all summarized in Table 1.

Optical emission spectra were taken of argon, nitrogen, and oxygen. Argon spectra from two pressures, 0.1 and 0.5 mTorr reveal that, as the pressure increases, the intensity of the excited ion peak (480.6 nm) decreases, while the excited neutral (750.4 nm) peak intensity remains relatively constant.

The energy level information needed to solve Equation 3 was obtained from a standard reference (18), which provided the necessary transition wavelength and energy information. For each gas studied, there are several possible de-excitation routes, and the wavelengths corresponding to these transitions were monitored while varying both the pressure and the incident microwave power. The results are presented in Figures 5a and 5b. As shown, the transition wavelength most sensitive to pressure change in the 10^{-4} Torr regime is the argon ion at 480.6 nm and the argon neutral at 750.4 nm. This same pair of transitions is also sensitive to changes in incident microwave power. Included in these figures is the ion saturation current, I_{sat}, measured in the Langmuir probe studies. McKillop, et al (14), have also studied Ar and O_2 and have reported both the most sensitive transition wavelengths and I_{sat} for the same pressure regime that we used, but for higher incident powers (700-2300 W). In both our studies and those of McKillop, the above transitions remain sensitive to changes in incident microwave power, and a relationship is seen between the emission intensity and I_{sat} when both pressure and power are varied. The latter observation indicates that the Ar^{+} 480.6 nm emission line intensity can provide a reasonable estimate of the relative concentration of Ar ions for the wide range of microwave powers used in these two studies.

A simulation was performed to explore the dependence of the electron temperature on the constant, C, and on the ion to neutral peak intensities. This provided a family of curves, as seen in Figure 6 for nitrogen, which gives a theoretical result for optical emission electron temperatures based on the use of Equation 3. This simulation is helpful in choosing a scaling constant which provides electron temperatures that are close in value to those from the probe. Also shown in Figure 6 is the experimental probe data, and when the theoretical curves are compared to the probe results, the best-fit value of C was 2 $eV^{-0.75}$. The curves match quite well until the negative of the natural log of the intensity ratio increases to 1.5 and above, which corresponds to pressures of $5x10^{-4}$ torr and above, confirming that the constant is pressure dependent.

Initially, argon plasmas were studied. These discharges are chemically inert and do not complicate the Langmuir probe measurements by reacting with the probe. Electron temperatures were measured at various pressures using Langmuir probes and with optical emission by monitoring the emission intensities of lines at 480.6 nm (Ar^+) ($E_g + E_{i,g}$ = 34.90 eV) and 750.3 nm (Ar^0) ($E_{p,g}$ = 13.42 eV) (18). For this pair of transitions, C was 0.2 $eV^{-0.75}$. The results, plotted in Figure 7a, show that both LP and OE techniques have the same trend across the 10^{-4} Torr pressure range.

The oxygen plasma emission lines at 464.9 nm (O^+) ($E_g + E_{i,g}$ = 39.16 eV) and 777.1-nm (O^0) ($E_{p,g}$ = 10.69 eV) (18) were monitored. When compared to probe results, C was 0.15 $eV^{-0.75}$, although this fit was not as good for the other two gases. The results obtained here are plotted in Figure 7b, and the optical emission technique, for the lower pressures of $1\text{-}2x10^{-4}$ Torr, resulted in electron temperatures of about 1-1.5 eV lower than those from the probe; at the higher pressures, both curves become flatter, with the electron temperatures obtained from OE 2-3 eV higher at first, and then within 1 eV of the probe results.

The nitrogen plasma emission lines at 467.8-nm (N_2^+) ($E_g + E_{i,g}$ = 40.75 eV) and 581.6 nm (N_2^0) ($E_{p,g}$ = 13.97 eV) (18) were monitored. The electron temperatures obtained with both methods agree quite well here, as seen in Figure 7c. For nitrogen, the electron temperatures for the $1.6\text{-}10x10^{-4}$ Torr range at applied power of 300 W are from 5-10 eV, and the plasma potentials range from 15 to 26 V. At a pressure of $2x10^{-4}$ Torr, as the applied power decreases to 100 W, the electron temperature decreases to 6.5 eV, demonstrating the importance of maintaining a low pressure environment if one is to achieve good efficiency in deposition processes. For argon, oxygen and nitrogen, the electron temperatures at an applied power of 300 W for the 10^{-4} Torr range are from 6-12 eV; the lower electron temperatures are found at lower pressures. The lower the pressure, the fewer gas molecules are present and the less chance the electrons have of colliding with another particle and losing energy. Thus, the longer lifetime results in the attainment of higher energies.

The dependence of the electron temperature on the value of this constant was also investigated. A small variation of C caused relatively small changes in the calculated electron temperature. For example, in the case of nitrogen, a variation of 0.5 $eV^{-0.75}$ in C resulted in an approximately 1 eV change in the optical emission calculated electron temperature.

VI. Conclusions

It is shown that optical emission is an extremely useful diagnostic tool due to its non-invasive nature. OE can be used to determine ionic and neutral species occurring in the plasma, and, with initial calibration using a probe, can yield reliable electron temperature measurements. It is very important to be certain of this calibration, which consists of finding a suitable constant to be used in conjunction with Equation 3 to relate the optical emission results to the initial probe results. An efficient way to accomplish this is to simulate the dependence of the optical emission electron temperature on the negative of the natural log of the ion to neutral intensity ratios while also varying C, and then to use the probe electron temperature to find the best fit. If the pressure regime over which one works is large, however, it may be necessary to choose a different C for the different pressure ranges.

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Figure 1: Schematic diagram of the experimental apparatus.

Figure 2a: Examples of Langmuir probe I-V curves from argon plasmas at (a) 1×10^{-4} and (b) 5×10^{-4} Torr.

Figure 2b: Examples of Langmuir probe ln(I)-V curves from argon plasmas at (a) 1×10^{-4} and (b) 5×10^{-4} Torr.

Figure 3: Ion density measurements for argon, nitrogen and oxygen as a function of pressure.

Figure 4: Plasma potentials for argon, nitrogen and oxygen as a function of pressure.

Figure 5a: Selected argon emission line intensities as a function of pressure. The lines subsequently used for the experiment were those that were the most sensitive to pressure changes. Also included is the ion saturation current measured in Langmuir probe studies.

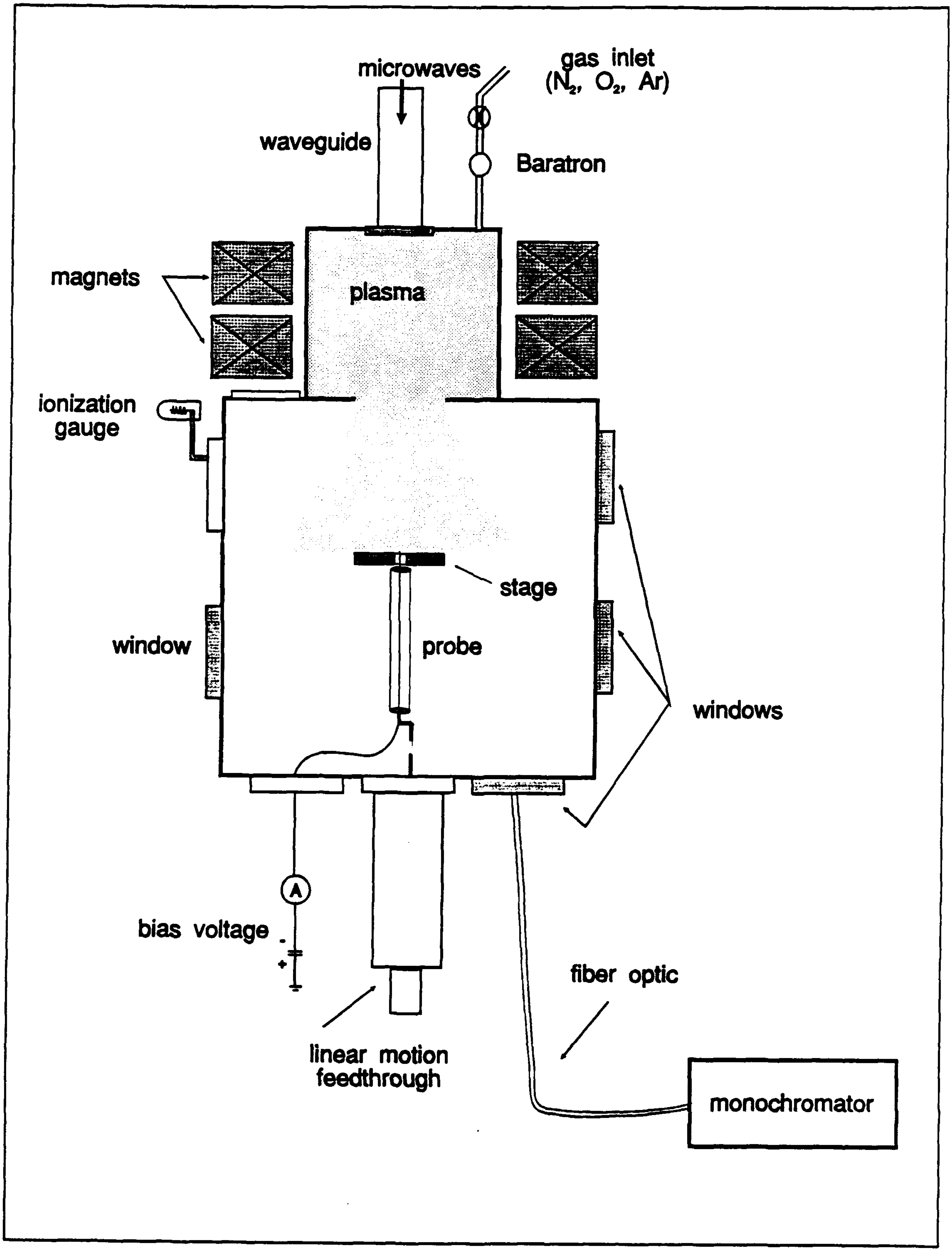
Figure 5b: Selected argon emission line intensities as a function of incident microwave power. The ion saturation current is higher than, but follows the same trend, as the intensity of the 480.6 nm Ar^+ ion emission, indicating that the emission intensity can be used as a reasonable estimate of the ion current.

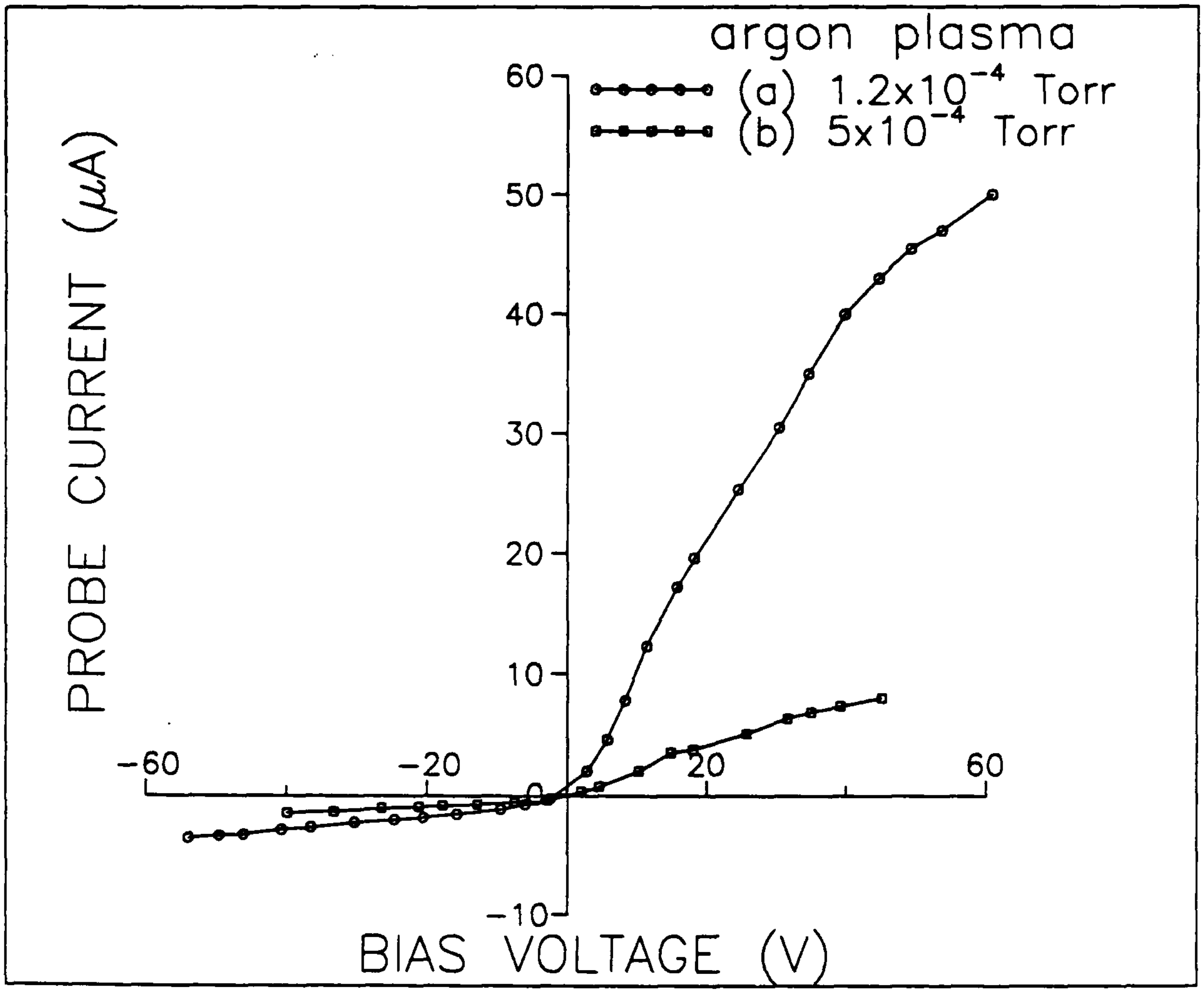
Figure 6: In the case of nitrogen, simulated calculations provide a family of curves, based on Equation 3, which illustrate the dependence of electron temperature on the ion to neutral intensity ratio as the value of the constant, C, is varied. Also shown are the Langmuir probe experimental electron temperatures. The value of C which best fits the experimental probe results to the simulated optical emission results is that of 2 $eV^{-0.75}$

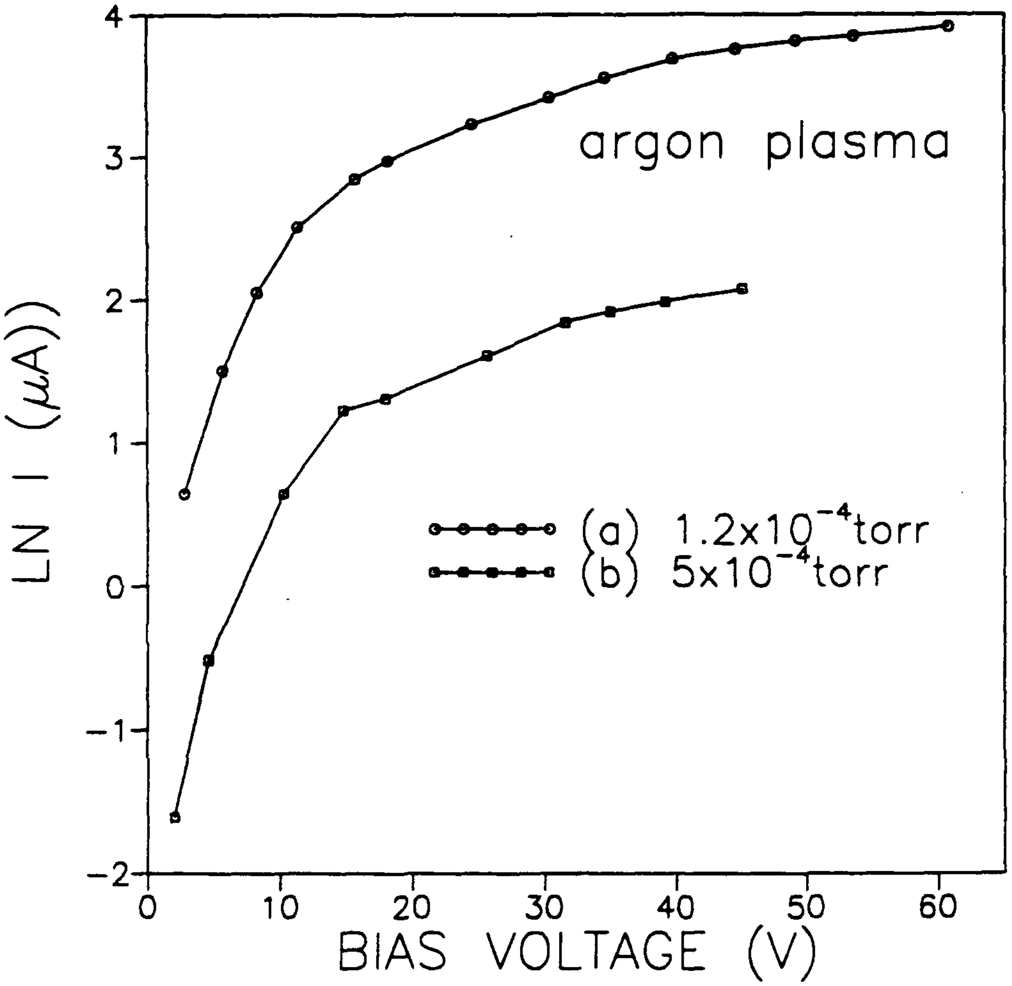
Figure 7: The dependence of electron temperature, as measured by both Langmuir probe and optical emission techniques, on pressure for (a) argon, (b) oxygen and (c) nitrogen.

List of Tables:

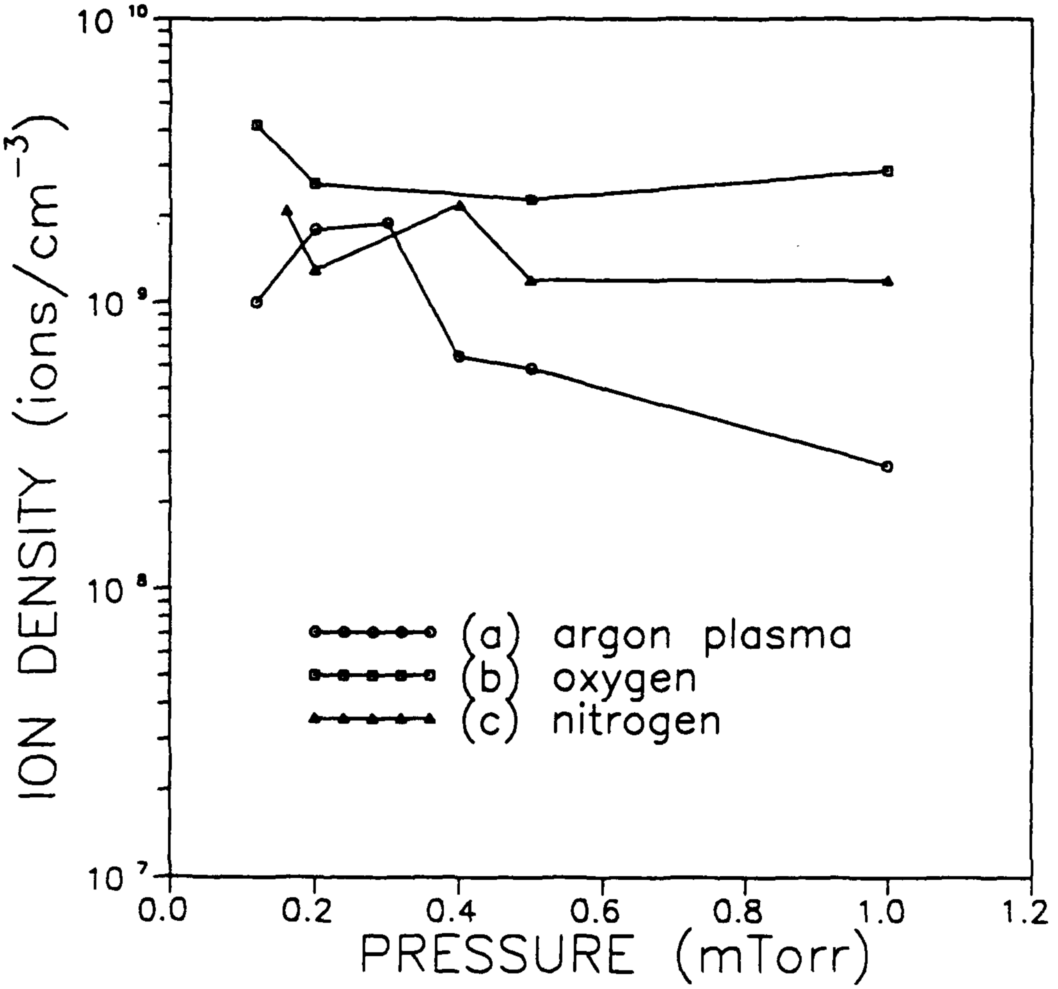
Pressure (mTorr)	argon				nitrogen				oxygen			
	N_i x10^9	V_p	T_e LP	T_e OE	N_i x10^9	V_p	T_e LP	T_e OE	N_i x10^9	V_p	T_e LP	T_e OE
.12	1.0	25	12.8	12.8	-	-	-	-	4.2	26	11.8	12.3
.16	-	-	-	-	2.1	26	10.6	10.5	-	-	-	-
.2	1.8	24	10.4	11.6	1.3	25	10	10.5	2.6	24	12	10.5
.3	1.9	22	10	8.7	-	-	-	-	-	-	-	-
.4	.65	18	9.7	6.0	2.4 2.0 1.1	24 24 25	8.4	8.7	-	-	-	-
.5	.59	18	7. 5	5.3	1.2 1.1	24 26	7.6	7.8	2.3	22	8	9.9
1.0	.27	17	6.2	4.3	1.2	17	6.8	6.3	2.9	18	6.8	7.8


microwaves
gas inlet
(N_2, O_2, Ar)
waveguide
Baratron
magnets
plasma
ionization
gauge
stage
window
probe
windows
bias voltage
linear motion
feedthrough
fiber optic
monochromator

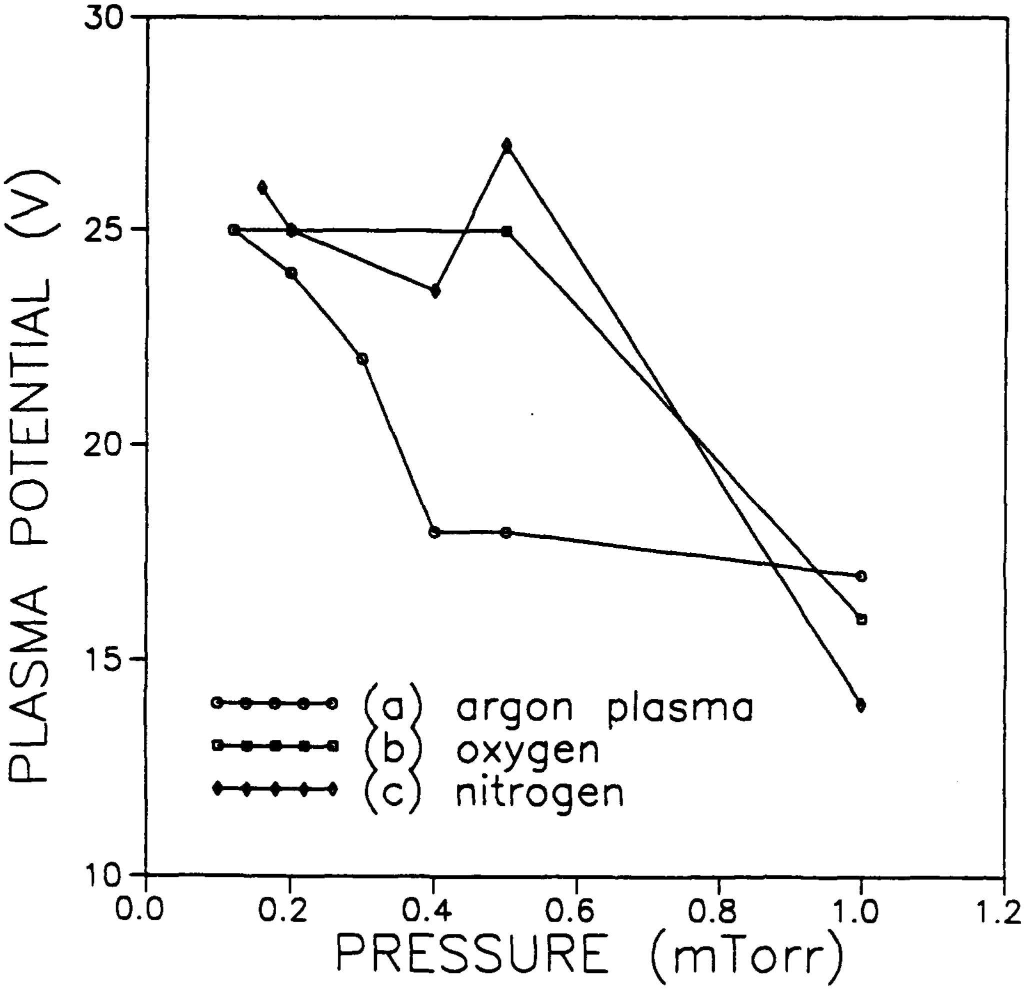

argon plasma
(a) 1.2x10^-4 Torr
(b) 5x10^-4 Torr
PROBE CURRENT (μA)
BIAS VOLTAGE (V)
60
50
40
30
20
10
0
-10
-60
-20
20
60



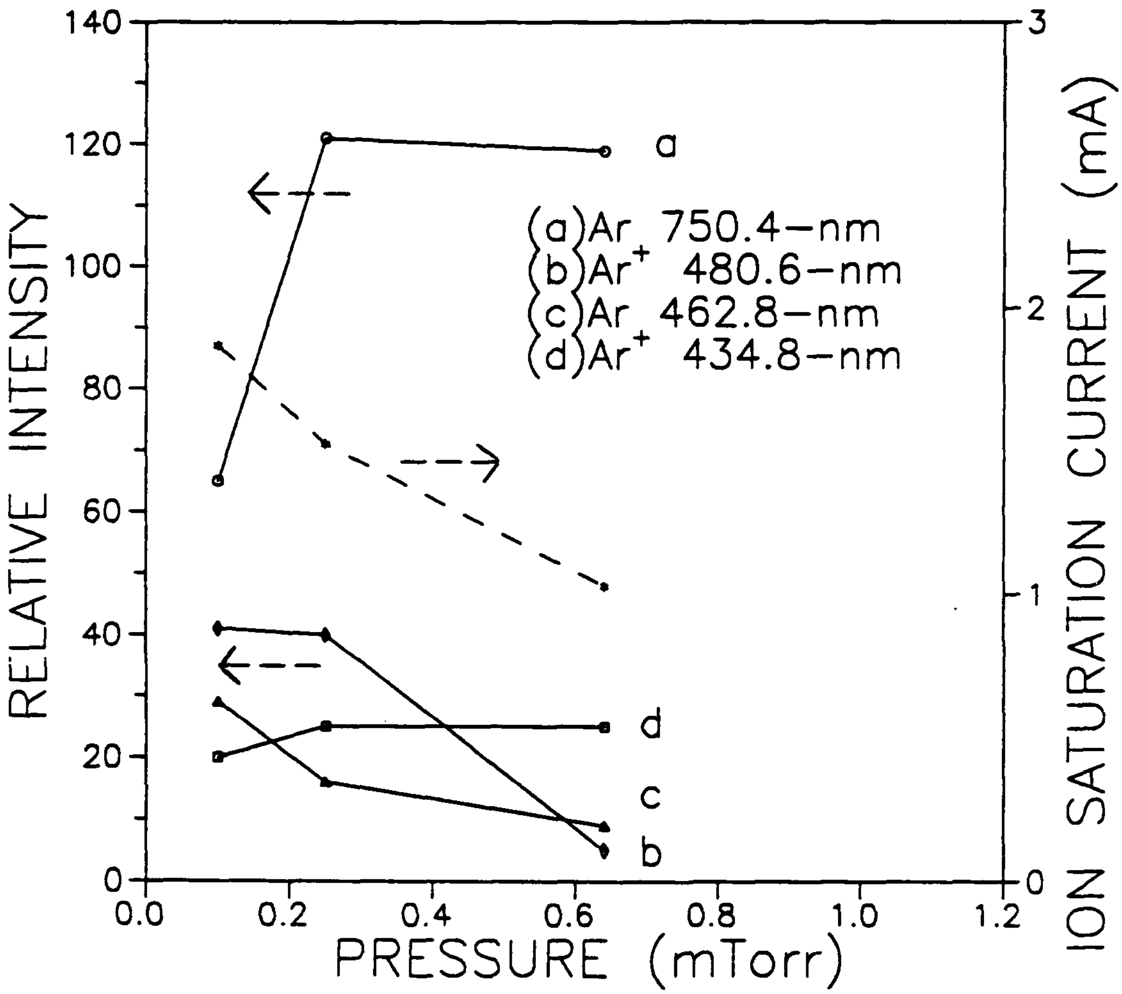
argon plasma
(a) 1.2×10^{-4}torr
(b) 5×10^{-4}torr
LN I (μA))
BIAS VOLTAGE (V)
4
3
2
1
0
−1
−2
0
10
20
30
40
50
60

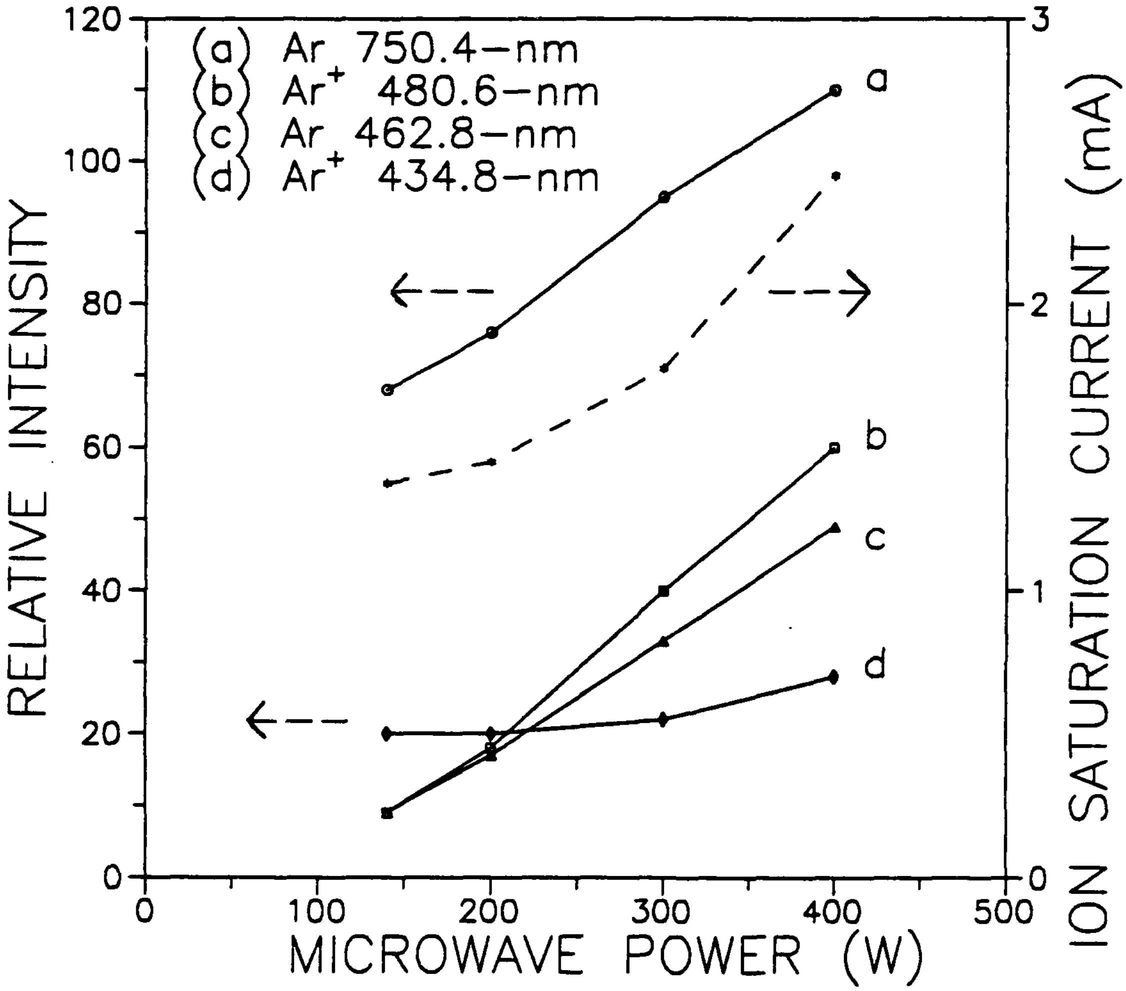


10 ¹⁰
10 ⁹
10 ⁸
10 ⁷
0.0
0.2
0.4
0.6
0.8
1.0
1.2
ION DENSITY (ions/cm⁻³)
PRESSURE (mTorr)
(a) argon plasma
(b) oxygen
(c) nitrogen



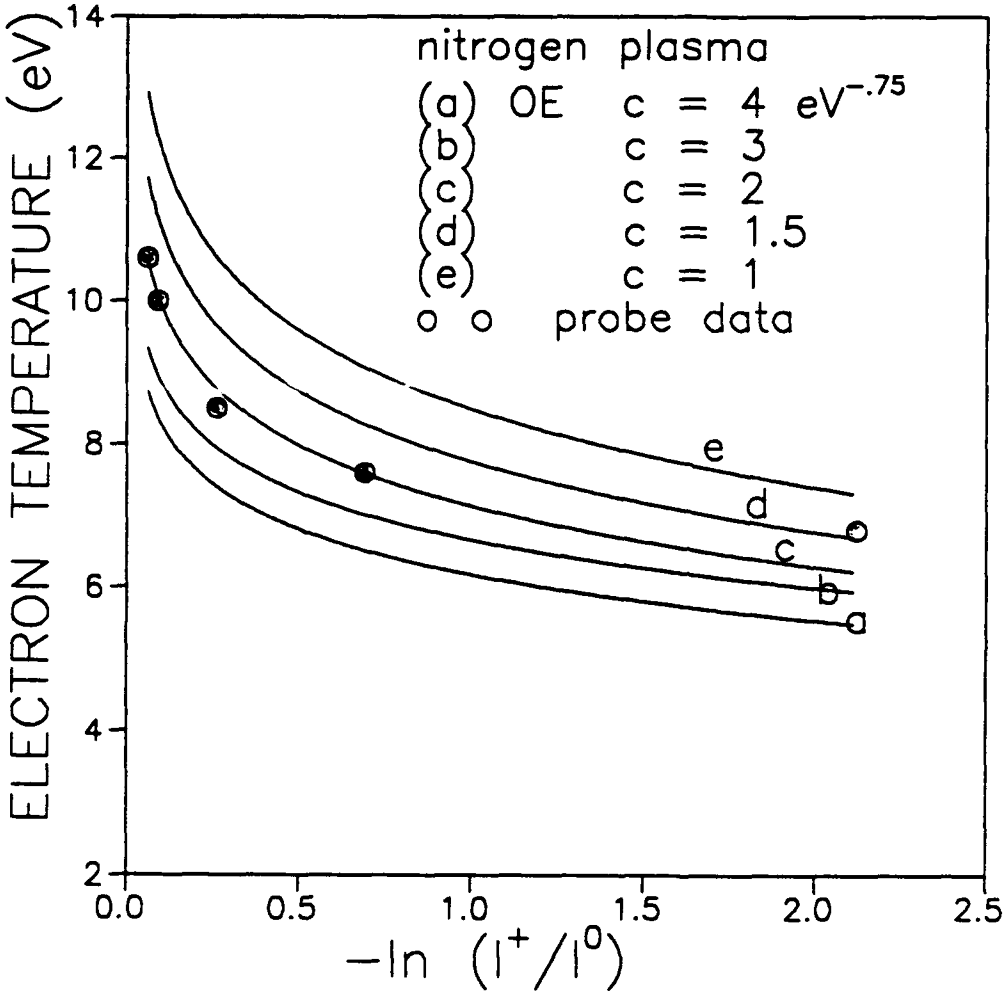
PLASMA POTENTIAL (V)
30
25
20
15
10
0.0
0.2
0.4
0.6
0.8
1.0
1.2
PRESSURE (mTorr)
(a) argon plasma
(b) oxygen
(c) nitrogen


140
120
100
80
60
40
20
0
3
2
1
0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
RELATIVE INTENSITY
ION SATURATION CURRENT (mA)
PRESSURE (mTorr)
a
b
c
d
(a)Ar 750.4-nm
(b)Ar+ 480.6-nm
(c)Ar 462.8-nm
(d)Ar+ 434.8-nm

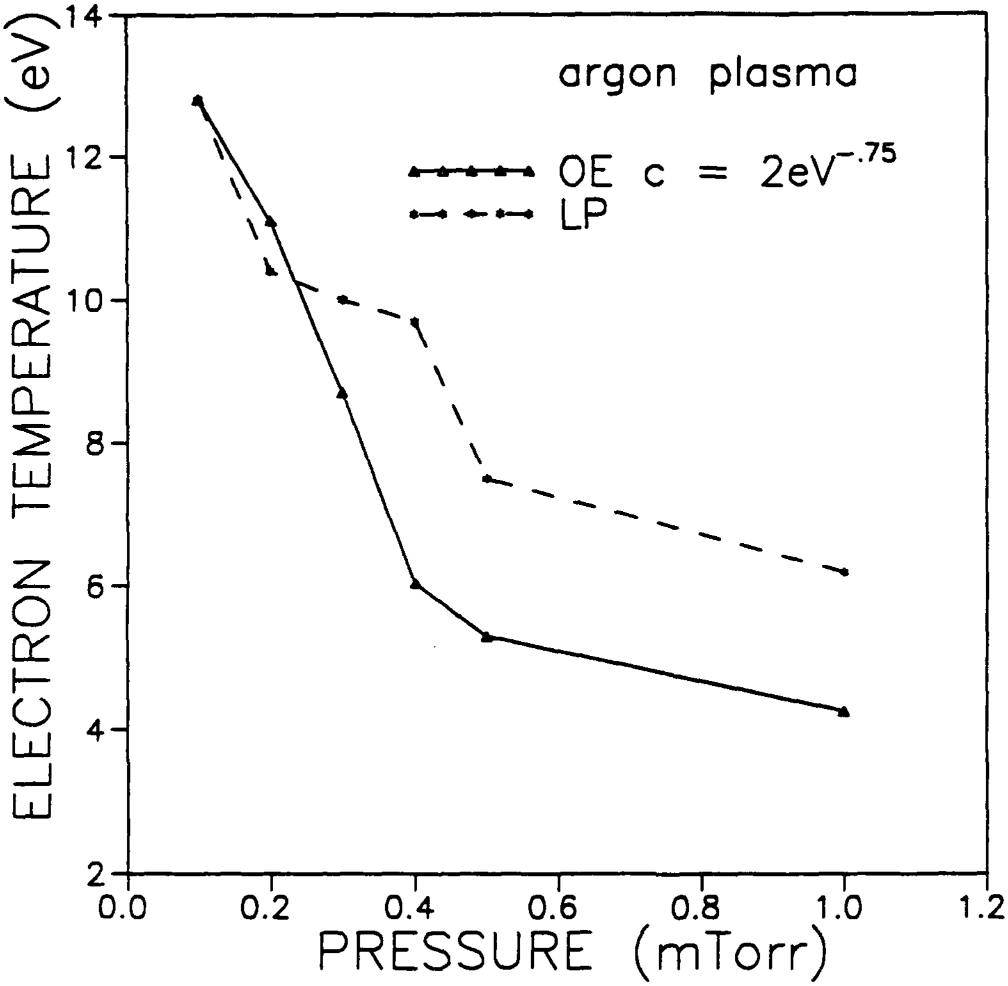


(a) Ar 750.4-nm
(b) Ar+ 480.6-nm
(c) Ar 462.8-nm
(d) Ar+ 434.8-nm
a
b
c
d
RELATIVE INTENSITY
ION SATURATION CURRENT (mA)
MICROWAVE POWER (W)
0
20
40
60
80
100
120
0
1
2
3
0
100
200
300
400
500

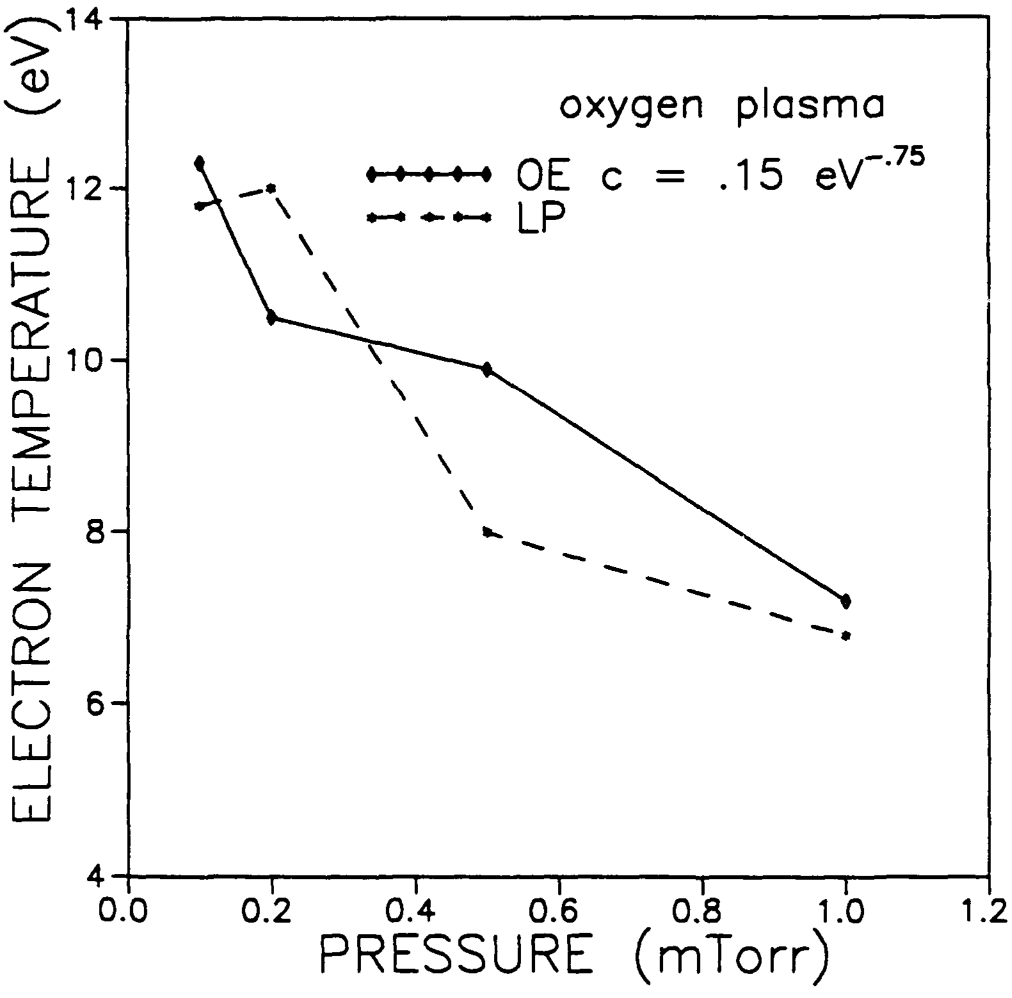
Fi



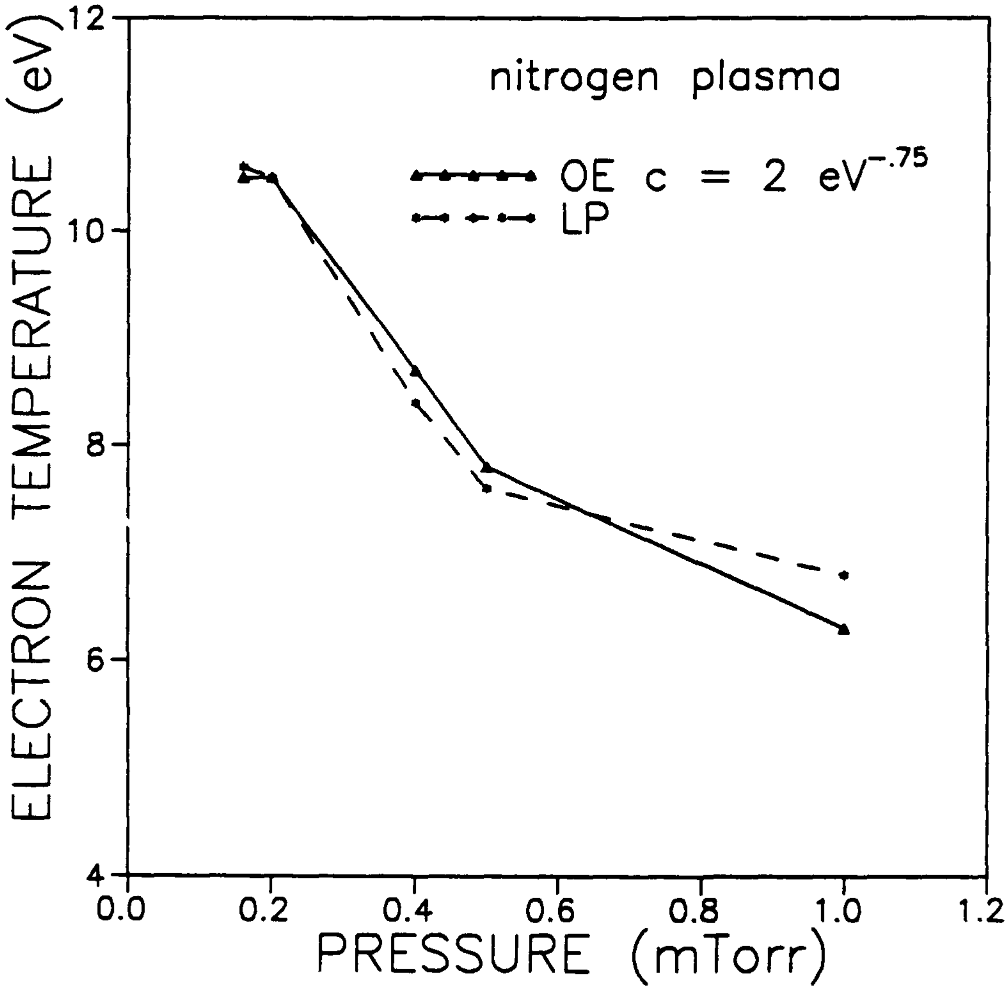
nitrogen plasma
(a) OE c = 4 eV^-.75
(b) c = 3
(c) c = 2
(d) c = 1.5
(e) c = 1
o o probe data
ELECTRON TEMPERATURE (eV)
−ln (I+/I0)
e
d
c
b
a
14
12
10
8
6
4
2
0.0
0.5
1.0
1.5
2.0
2.5



argon plasma
OE c = 2eV^-.75
LP
ELECTRON TEMPERATURE (eV)
14
12
10
8
6
4
2
0.0
0.2
0.4
0.6
0.8
1.0
1.2
PRESSURE (mTorr)


oxygen plasma
OE c = .15 eV^-.75
LP
ELECTRON TEMPERATURE (eV)
PRESSURE (mTorr)
14
12
10
8
6
4
0.0
0.2
0.4
0.6
0.8
1.0
1.2

7c



nitrogen plasma
OE c = 2 eV$^{-.75}$
LP
ELECTRON TEMPERATURE (eV)
PRESSURE (mTorr)
12
10
8
6
4
0.0
0.2
0.4
0.6
0.8
1.0
1.2